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मानक

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IS 5346 (1994): Synthetic Food Colour - Preparations and Mixtures [FAD 8: Food Additives]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 5346 : 1994
(Reaffirmed 2004)

भारतीय मानक
संश्लिष्ट खाद्य रंग— निमित्तियाँ और मिश्रण— विशिष्टि
(दूसरा पुनरीक्षण)

Indian Standard
**SYNTHETIC FOOD COLOUR—PREPARATIONS AND
MIXTURES—SPECIFICATION**
(Second Revision)

Second Reprint JANUARY 2006
(Including Amendment No. 1)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1994

Price Group 3

AMENDMENT NO. 1 FEBRUARY 2005
TO
IS 5346 : 1994 SYNTHETIC FOOD COLOUR —
PREPARATIONS AND MIXTURES — SPECIFICATION
(Second Revision)

[Page 1, clause 4.4(i)] — Substitute 'Potable water (conforming to IS 10500 : 1991) or Packaged drinking water (conforming to IS 14543 : 2004)' for 'Potable water (conforming to IS 10500 : 1991)'

(Page 2, Table 1) — Substitute 'IS 1699 : 1995' for 'IS 1699 : 1974'

(Page 2, clause 6.1, line 4 and 5) — Substitute '4 of IS 1699 : 1995' for '4 of IS 1699 : 1994'

(Page 3, Annex A) — Substitute '1699 : 1995 Methods of sampling and test for food colours (second revision)' for '1699 : 1994 Methods of sampling and test for food colours (second revision)'

(FAD 8)

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**AMENDMENT NO. 2 FEBRUARY 2007
TO
IS 5346 : 1994 SYNTHETIC FOOD COLOUR—
PREPARATIONS AND MIXTURES —
SPECIFICATION**

(Second Revision)

[Page 1, *clause 4.4(n)*] — Substitute 'Common salt (conforming to IS 253 : 1985) or Iodized Salt (conforming to IS 7224 : 1985)' for 'Edible common salt (conforming to IS 253 : 1985)'.

(Page 3, *Annex A*) — Insert the following at the appropriate place.

<i>'IS No</i>	<i>Title</i>
7224 : 1985	Specification for Iodized salt (<i>first revision</i>)'

(FAD 8)

AMENDMENT NO. 3 MARCH 2010
TO
IS 5346 : 1994 SYNTHETIC FOOD COLOUR —
PREPARATIONS AND MIXTURES —
SPECIFICATION

(Second Revision)

(Page 2, clause 4.7.1) — Substitute the following for the existing:

‘The product shall be processed, packed, stored and distributed under hygienic conditions in licensed premises as per IS 2491.’

(FAD 8)

Reprography Unit, BIS, New Delhi, India

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

This Standard was first issued in 1969. It was revised in 1975 with a view to bringing the standard up to date and to align it with the specifications for basic synthetic food colour issued by the FAO/WHO. In that revision the scope was widened to cover synthetic food colour mixtures, as well as suitable quality of diluents and filler materials to be used for manufacture of synthetic food colour preparations. Requirements for mixed oxides and copper were deleted.

This standard is being revised again taking into consideration the specifications laid down under EEC Directives and Canadian Food Laws. This revision also incorporates the five amendments issued to the earlier revision as well as method of test for dye content in food colour preparations which was earlier published as IS 6120. In this revision, the tolerances of estimated total dye content from the declared value had been reviewed with a view to reduce these. However, on the basis of data collected, the tolerances in the case of powder preparations were retained at the earlier levels, whereas in the case of liquid preparations, the negative tolerance was reduced from the earlier value of 15 percent but the tolerance on the positive side was retained at the earlier level.

A limited number of synthetic food colours have been permitted under the *Prevention of Food Adulteration Rules, 1955*. Specifications for these basic synthetic food colours have already been issued by the Bureau. A number of combinations of these colours are prepared with or without diluents and preservatives so as to develop different shades. This standard specifies the requirements of such synthetic food colour preparations or mixtures which are meant to be used for imparting colour to foods.

In the preparation of this standard, due consideration has been given to the Rules prescribed by the Government of India under *Prevention of Food Adulteration Act, 1954*. These rules, *inter-alia* prescribe:

‘All food colours including natural colouring matter and permitted synthetic colours and their preparations or mixtures excluding saffron and curcumin shall be sold only under the BIS Certification Mark.’

Due consideration has also been given to the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977*. The standard is, however, subject to restrictions imposed under these wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (revised)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SYNTHETIC FOOD COLOUR — PREPARATIONS AND MIXTURES — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for synthetic food colour preparations and synthetic food colour mixtures.

2 REFERENCES

The Indian Standards given at Annex A are necessary adjuncts to this standard.

3 TERMINOLOGY

3.0 For the purpose of this standard, the following definitions shall apply.

3.1 Colour Preparation

A preparation containing one or more of the permitted synthetic food colours along with diluents and/or filler materials and meant to be used for imparting colour to foods.

3.2 Mixture

A mixture of two or more permitted synthetic food colours without diluents and filler materials and meant to be used for imparting colour to foods.

4 REQUIREMENTS

4.1 Permitted synthetic food colours used in the colour preparations or in mixtures shall conform to the relevant specifications prescribed by the Bureau of Indian Standards.

4.2 The colour preparations could be either in the form of a liquid or powder. Powder preparations shall be reasonably free from lumps and any visible extraneous/foreign matter. Liquid preparations shall be free from sediments.

4.3 Colour preparations or mixture may contain preservatives and stabilizers permitted under *Prevention of Food Adulteration Rules, 1955*.

4.4 Only the following diluents or filler materials shall be permitted to be used in colour preparations:

- i) Potable water (conforming to IS 10500 : 1991)
- ii) Edible common salt (conforming to IS 253 : 1985)
- iii) Sugar (Conforming to IS 1679 : 1960)
- iv) Dextrose monohydrate (conforming to IS 874 : 1992)
- v) Liquid glucose (conforming to IS 873 : 1974)
- vi) Sodium sulphate (conforming to IS 255 : 1982)

- vii) Tartaric acid (conforming to IS 880 : 1956)
- viii) Glycerine (conforming to IS 1796 : 1986)
- ix) Propylene glycol, food grade (conforming to IS 13702 : 1993)
- x) Acetic acid, dilute (conforming to IS 695 : 1986)
- xi) Sorbitol, food grade (conforming to IS 4750 : 1968)
- xii) Citric acid (conforming to IS 13186 : 1991)
- xiii) Sodium carbonate and sodium hydrogen carbonate
- xiv) Lactose (conforming to IS 1000 : 1989)
- xv) Ammonium, sodium and potassium alginates (conforming to IS 5191 : 1993)
- xvi) Dextrins
- xvii) Ethyl acetate
- xviii) Starches
- xix) Diethyl ether
- xx) Ethanol
- xxi) Glycerol mono, di and tri acetate
- xxii) Edible oils and fats
- xxiii) Isopropyl alcohol (conforming to IS 11686 : 1986)
- xxiv) Bees wax (conforming to IS 1504 : 1974)
- xxv) Sodium and ammonium hydroxide
- xxvi) Lactic acid (conforming to IS 9971 : 1981)
- xxvii) Carragenan and gum arabic (conforming to IS 6795 : 1972) (for annatto only)
- xxviii) Gelatin (conforming to IS 5719 : 1970)
- xxix) Pectin

4.5 Total Dye Content

The total synthetic dye content, percent by mass (m/v) in the colour preparation or in the mixture shall be declared on the label of the container [see 5.2.1 (c)]. In powder preparations the declared value shall be on moisture-free basis and in case of liquid preparations on as-in basis. When determined by the method prescribed in Annex B, the total dye content shall be within the tolerance limits given below on the declared value :

- a) Liquid preparations + 15 percent
 - 5 percent
- b) Solid preparations \pm 7.5 percent

4.6 The limits of impurities shall be as given in Table 1.

Table 1 Limits for Impurities
(Clause 4.6)

Sl No.	Characteristics	Requirement	Method of Test, Ref to Clause of IS 1699 : 1974
(1)	(2)	(3)	(4)
i)	Water insoluble matter, percent by mass (on dry basis), <i>Max</i>	1.0	7
ii)	Lead (as Pb), mg/kg, <i>Max</i>	10	15
iii)	Arsenic (as As), mg/kg, <i>Max</i>	3.0	15
iv)	Heavy metals, mg/kg, <i>Max</i>	40	16

4.7 Freedom from Contaminants

Precautions shall be taken to ensure that the material is free from mercury, copper and chromium in any form; aromatic amines, aromatic nitro compounds, aromatic hydrocarbons, polycyclic aromatic hydrocarbon, 2-naphthyl amine/benzidine, amino-4-diphenyl (xenylamine) or their derivatives and cyanides.

4.7.1 The product shall be processed, packed, stored and distributed under hygienic conditions in licenced premises (see IS 2491 : 1972).

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in glass, metal or polyethylene containers, or cardboard containers suitably lined with polyethylene or any other suitable containers as agreed to between the purchaser and the seller

NOTE - Preparations containing indigo carmine are known to deteriorate during storage. Manufacturers should, therefore, take precautions in their formulation and packing

5.2 Marking

5.2.1 Each container shall be legibly and indelibly marked with the following information:

- The words 'Synthetic Food Colour Preparation' or 'Synthetic Food Colour Mixture',
- The names and colour index, number of the various food colours and names of other ingredients used in the preparation;
- Percentage of total synthetic dye content (4.5);
- List of ingredients in descending order of composition;
- Source of manufacture;
- Date of manufacture;

g) Net mass in g or kg, or litre or ml;

h) Batch or code number;

j) Expiry date in case of liquid synthetic food colour preparations or mixtures containing indigo carmine, and

k) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955.*

5.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material for tests shall be drawn and criteria for ascertaining conformity to the requirements of this specification shall be determined according to the method prescribed in 4 of IS 1699 : 1994

7 TESTS

7.1 Tests shall be carried out as prescribed in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Clause 2)

LIST OF REFERRED INDIAN STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
253 : 1985	Edible common salt (<i>third revision</i>)	1796 : 1986	Glycerine (<i>second revision</i>)
255 : 1982	Sodium sulphate, anhydrous (technical grade) (<i>second revision</i>)	2491 : 1972	Code for hygienic conditions for food processing units (<i>first revision</i>)
695 : 1986	Acetic acid (<i>third revision</i>)	4750 : 1968	Sorbitol, food grade
873 : 1974	Liquid glucose (<i>first revision</i>)	5191 : 1993	Sodium alginate, food grade (<i>first revision</i>)
874 : 1992	Dextrose monohydrate (<i>third revision</i>)	5719 : 1970	Gelatin, food grade
880 : 1956	Tartaric acid	6795 : 1972	Acacia (<i>Arabic</i>) gum, food grade
1000 : 1989	Lactose, commercial (<i>first revision</i>)	9971 : 1981	Lactic acid, food grade
1070 : 1992	Reagent grade water (<i>third revision</i>)	10500 : 1991	Drinking water (<i>first revision</i>)
1504 : 1974	Beeswax (<i>second revision</i>)	11686 : 1986	Iso-propyl alcohol, food grade
1679 : 1960	Sugar used in food preservation industry	13186 : 1991	Citric acid, food grade
1699 : 1994	Methods of sampling and test for food colours (<i>second revision</i>)	13702 : 1993	Propylene glycol, food grade

ANNEX B

(Clause 4.5)

DETERMINATION OF TOTAL DYE CONTENTS

B-1 PRINCIPLE

B-1.1 The component dyes of the food colour preparations are separated and identified by paper chromatography and these components are then estimated by spectrophotometric methods, either:

- by finding out the absorbancy of the individual component dyes at their absorption maxima, after quantitative elution from the paper chromatogram; or
- by direct estimation of the absorbancy, at some selected wavelengths, depending on the nature of the individual component dye.

B-2 REAGENTS

B-2.1 Chromatographic Paper

Rectangular sheets (32 x 19.5 cm) of Whatman No. 1 or equivalent filter paper. Nine slots (5 x 24 cm) should be cut from the paper, parallel to the long side and at a distance of 2 cm from one of the short edge, so as to

have 10 strips, 1.5 cm wide, joined at the top and bottom.

B-2.2 Chromatographic Solvents

B-2.2.1 *Solvent No. 1* - 1 ml of ammonium hydroxide (sp gr 0.91) + 99 ml water.

B-2.2.2 *Solvent No. 2* - Isobutanol : ethanol : water (1 : 2 : 1).

B-2.2.3 *Solvent No. 3* - *n*-butanol : water : glacial acetic acid (20 : 12 : 5).

B-2.2.4 *Solvent No. 4* - 10 g trisodium citrate + 50 ml ammonium hydroxide (sp gr 0.91) + 50 ml water.

B-2.3 Solvents for Elution

B-2.3.1 *Hydrochloric Acid* - in 70 percent ethanol 0.1 N.

B-2.3.2 *Hydrochloric Acid* - 0.1 N.

B-2.3.3 *Sodium Hydroxide Solution* - 0.1 N.

B-2.4 *Standard Food Colours* - conforming to relevant Indian Standards.

B-3 APPARATUS**B-3.1 Chromatographic Tank**

All glass chromatographic tank, 50 x 30 x 25 cm, suitable for both ascending and descending chromatography.

B-3.2 Spectrophotometer

A reliable spectrophotometer, fitted with photomultiplier or phototube with amplifier, and glass-cells having 1.00 cm light path.

B-3.3 Micro-Pipette or Algh-Micro-Syringe**B-4 PROCEDURE****B-4.1 Identification****B-4.1.1 Spotting**

Prepare aqueous solution (1 mg/ml) of the food colour preparations. Spot accurately measured quantities (20 μ to 40 μ) on the 10 strips of the chromatographic paper (B-2.1), at points 0.5 cm above the line joining the lower ends of the slots, that is, at 2.5 cm from the edge.

B-4.1.2 Preparation of Chromatographic Tank

B-4.1.2.1 Set up the all-glass chromatographic tank (3.1) at a place, free from any vibration. Hang from one of the troughs (at the top) a filter paper dummy, 35 x 20 cm at the inner side; cut some serrations along the full length at the bottom of the dummy paper to allow easy and uniform dripping of the solvent. Keep the trough always filled with solvent during chromatography by adding solvent through the corresponding hole in the lid. Fix a glass rod, with a bent hook at the bottom, with a rubber plastic stopper, through a hole of the cover near the centre and at a distance of about 3 cm from the plane of the dummy paper; attach the glass rod in such a way so that it is possible to push it up and down, without causing any vibrations to the tank.

B-4.1.3 Chromatography

B-4.1.3.1 Pour about 750 ml of the solvent to be used (B-2.2), inside the tank. Fill the trough with dummy paper also with the same solvent. The solvent would start to soak the dummy paper and descend. Attach after an hour the spotted chromatographic paper at the top to a flat metal strip, 200 x 15 x 1.5 mm (approximately), with a central hole for the hook. Suspend this from the hook of the glass rod, inside the tank. Allow the chromatographic paper to get saturated in the closed chamber for two and a half hour. Push the paper down with the help of the suspending glass rod, so that about 5 mm of the lower edge of the chromatographic paper dips in the solvent below.

The solvent would gradually ascend the paper. When the solvent reaches about 1 cm below the line joining the upper end of the slots, remove the paper carefully, mark the solvent line immediately with a pencil and allow to dry in the air.

B-4.2 Quantitative Estimation**B-4.2.1 Determination of Pure Dye Content of Standard Food Colours**

The pure dye content of each food colour, having purity

according to the relevant Indian Standard specification shall be estimated by titanium trichloride reduction method, prescribed in the Indian Standards for each individual food colour.

B-4.2.2 Determination of Absorption Spectrum of Standard Food Colours

Weigh/measure accurately 100 mg/100 ml of each of the standard food colours separately. Dissolve in redistilled water. Make from these stock solutions, solutions of the dyes in water with concentration approximately 1 mg/100 ml. Also make the solutions in 0.1 N hydrochloric acid, 0.1 N hydrochloric acid in 70 percent alcohol and 0.1 N sodium hydroxide. Find out absorption spectra of these solutions in the range 420 to 650 m μ , using cells of 1.00 cm light path. From these absorption spectra, calculate extinction coefficient ($E^{1\%}_{1\text{cm}}$) at absorption maxima on the basis of pure dye contents (B-4.2.1).

B-4.2.3 Separation and Elution Method

B-4.2.3.1 From the chromatogram of the food colour preparation (B-4.1.3.1) cut the separated bands of individual colours carefully and elute with 0.1 N hydrochloric acid in 70 percent ethanol or with other suitable eluting solutions (B-2.3). Make up the elute to 25 ml. Find out optical density of these elutes at respective wavelengths of absorption maxima (B-4.2.2), using cells of 1.00 cm light path. Use the extracts of equivalent portion from the blank part of the chromatogram in the same solvent as 'blank' in the optical density determination.

B-4.2.3.2 Calculate from these optical densities, the amounts of individual component colours present in the food colour preparation using the extinction coefficients ($E^{1\%}_{1\text{cm}}$) of the respective standard colour (B-4.2.2) given below. Compute the amounts together to find out the total dye content of the food colour preparations:

Amount of a dry component in a food colour preparation (g/100 g of food colour preparation)

$$= \frac{OD}{E^{1\%}_{1\text{cm}}} \times \frac{100}{C}$$

where

OD = the observed optical density at absorption maxima of the individual component, separated and eluted;

$E^{1\%}_{1\text{cm}}$ = extinction coefficient of the standard sample of the same by content, in the same solvent; and

C = equivalent concentration of the food colour preparation per 100 ml of the final solution.

NOTES

1 Following three major factors shall be taken into consideration for calculation of 'C':

- Concentration of original food-colour solution for chromatography, which should be approximately 1 mg/ml (B-4.1.1).
- Amount of dye solution spotted, which should be 20 μ to 40 μ (B-4.1.1).

- c) Final volume of the elute (which should be about 25 ml, but may have to be varied according to the intensity of colour), to be used for measuring OD (B-4.2.3.1).

2 This method shall not be applicable for determining indigotine, which might be present in some food colour preparations. For its determination direct spectrophotometric method (B-4.2.4) should be used.

B-4.2.4 Direct Spectrophotometric Method

B-4.2.4.0 Some food colours, like indigotine and erythrosine, are unstable in paper chromatogram and should be directly estimated by suitable optical methods. Moreover, as the elution method (B-4.2.3) requires several manipulative steps, there might be some difference in the results of duplicate estimations. By direct spectrophotometric method, this can be avoided.

B-4.2.4.1 Principle

From the absorption spectra of the standard food colours (B-4.2.2), ratios of OD (optical density) of a particular dye at wavelength maxima and minima of other dyes, to the OD at its wavelength maxima are calculated. For example, for tartrazine, it shall be necessary to find out:

$$\frac{E_{485}}{E_{430}}, \frac{E_{505}}{E_{430}}, \frac{E_{516}}{E_{430}}, \frac{E_{520}}{E_{430}}, \frac{E_{560}}{E_{430}}, \frac{E_{610}}{E_{430}}$$

where

E_{430} = OD for tartrazine at the wavelength of maximum absorption and $E_{485}, E_{505}, E_{516}, E_{520}, E_{560}, E_{610}$ are respectively ODs of tartrazine at wavelength maxima of sunset yellow, Ponceau 4R, amaranth, carmoisine sunset yellow (nunina) and indigotine.

B-4.2.4.2 Procedure

Dissolve accurately weighed quantity of the food colour preparation in water and then appropriately dilute with water or 0.1 N hydrochloric acid or 0.1 N sodium hydroxide, to give a final concentration of about 1 mg/100 ml. Determine the optical densities of this final diluted solution at the wavelength maxima of the component dyes in this food-colour preparation, as revealed by chromatography (B-4.1). In case of the mixture of Ponceau 4R with carmoisine and amaranth, OD values are found out, in 0.1 N sodium hydroxide at appropriate fixation points [B-4.2.4.3 (c)].

B-4.2.4.3 Calculation

- In case of mixtures, where one of the components has got an absorption maxima at a wavelength, where other components have little or no absorption, the value shall be directly calculated, after necessary correction, as in the case of mixture of tartrazine and indigotine, a mixture of sunset yellow and carmoisine or similar other mixtures.
- In case, where each of the components of a mixture, has got some optical absorption at the wavelength maxima of other components, the value shall be calculated using the following

formula:

$$x + b_1 y + c_1 z = OD_1$$

$$a_1 x + y + c_2 z = OD_2$$

$$a_2 x + b_2 y + z = OD_3$$

where

x, y, z are the corrected OD of the three components at their wavelength maxima, OD_1, OD_2, OD_3 are the observed ODs at the three wavelength maxima; and a_1, a_2, b_1, b_2 and c_1, c_2 are ratios of OD at the wavelength maxima of the other components to the OD of the particular components at its wavelength maxima (B-4.2.2). Calculate from x, y and z the concentration of the respective colour components.

- c) In case of mixtures of Ponceau 4R with carmoisine calculate according to the following equation:

$$\frac{E \lambda_1 - (E \lambda_2 - E \lambda_3) \frac{\lambda_3 - \lambda_1}{\lambda_3 - \lambda_2} - x}{E \lambda_3 - x} = k$$

where

λ_1 = wavelength of absorption maximum for carmoisine,

λ_2 and λ_3 = wavelengths on either side of where OD for the particular dye are equal,

$E \lambda_1, E \lambda_2$ = observed ODs in 0.1 N sodium hydroxide at λ_1, λ_2 and λ_3 ,

k = ratio of OD of the particular pure dye at λ_1 and λ_2 , and

x = unknown which may be solved from the above equation.

When x is known, the true OD at maxima for the particular dye, carmoisine present in the mixture can be calculated from the numerator of the above equation, namely:

$$E_{(Cord)} = E \lambda_1 - (E \lambda_2 - E \lambda_3) \frac{\lambda_3 - \lambda_1}{\lambda_3 - \lambda_2} - x$$

From the corrected OD, the amount of amaranth or carmoisine present in the mixture may be calculated from extinction coefficient, $E_{1\%}^{1\text{cm}}$. Subtracting the contribution of carmoisine from the observed OD, the amount of Ponceau 4R may be found out.

NOTES

- Only three readings are necessary for all the calculations.
- Values for fixation point for carmoisine in 0.1 N sodium hydroxide were found to be

Value	Carmoisine
500 mμ	
490 mμ	
517.5 mμ	
1 040	

B-4.2.5 The test report shall indicate which of the two methods (B-4.2.3 or B-4.2.4) has been employed for quantitative determination of dyes in food colour preparations.

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